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(54) **METHODE POUR PREPARER DES PREMELANGES DE
POLYMERE DE SILICONE ET DE SOLIDE, A HAUTE
TENEUR EN CHARGE**
(54) **PROCESS FOR THE PREPARATION OF SILICONE POLYMER/
SOLID PREMIXES HAVING A HIGH FILLER CONTENT**

(57) Prémélange de polymère de silicone et d'un solide, préparé par incorporation dans (A) un mélange renfermant (a) 50 à 100 parties en poids d'au moins un polyorganosiloxane renfermant jusqu'à 5 % en moles de groupes réticulables, et (b) 5 à 200 parties en poids d'au moins un composé organosilicié volatil, ne renfermant pas de groupe hydrolysable, 100 à 500 parties d'un solide (B), de façon à obtenir une composition; le composé organosilicié est ensuite séparé (b) de cette composition (b); enfin, cette dernière est soumise à une contrainte mécanique.

(57) A silicone polymer/solid premix may be prepared by incorporating into (A) a mixture comprising (a) 50 to 100 parts by weight of at least one polyorganosiloxane containing up to 5 mol % of crosslinkable groups, and (b) 5 to 200 parts by weight of at least one readily volatile organosilicon compound containing no hydrolyzable groups, 100 to 500 parts by weight of (B) a solid, thereby forming a composition; removing (b) said organosilicon compound (b) from said composition; and then subjecting said composition to mechanical stress.



PROCESS FOR THE PREPARATION OF SILICONE
POLYMER/SOLID PREMIXES HAVING
A HIGH FILLER CONTENT

ABSTRACT

A silicone polymer/solid premix may be prepared by incorporating into (A) a mixture comprising (a) 50 to 100 parts by weight of at least one polyorganosiloxane containing up to 5 mol % of crosslinkable groups, and (b) 5 to 200 parts by weight of at least one readily volatile organosilicon compound containing no hydrolyzable groups, 100 to 500 parts by weight of (B) a solid, thereby forming a composition; removing (b) said organosilicon compound (b) from said composition; and then subjecting said composition to mechanical stress.

TITLE OF THE INVENTIONPROCESS FOR THE PREPARATION OF SILICONE POLYMER/SOLID
PREMIXES HAVING A HIGH FILLER CONTENTBACKGROUND OF THE INVENTIONField of the Invention:

The invention relates to a process for the preparation of silicone polymer/solid premixes having a high filler content which are employed for the preparation of silicone rubbers.

Description of the Background:

Processes for distributing solids in liquids are known. The solid is usually incorporated into the liquid with the aid of mixing organs which rotate at more or less rapid speeds, and as a result the solid is wetted completely. The resulting mixture of wetted solid and liquid is then subjected to mechanical stress in a mixing chamber for a sufficiently long time to achieve as homogeneous as possible a distribution of the solid in the liquid.

To intensify this mechanical stress, a procedure is often followed (Masterbatch process, cf. Degussa "Schriftenreihe Pigmente" [Pigments Publication Series] Number 63, 1995) which incorporates the total amount of a solid initially in only a portion of the liquid. As a result of the higher consistency of the solid/liquid mixture, which can be achieved via a higher solids content, kneading and/or shear energy can be introduced considerably more effectively by a mixing organ, since the introduction of mixing energy has a greater effect on regions of the goods to be mixed which are close to the mixing organ. After mechanical stress on the mixture of solid and the portion of liquid, the mixture is diluted with further liquid.

Incorporation of a large amount of solids into liquids by the masterbatch process presents problems. It is irrelevant here whether the process is carried out in an open mixing unit, such as a twin-shaft kneader or dissolver, or in a closed mixer, such as an internal mixer or compression mixer, or in a continuous mixer, for example in a twin-screw extruder. If too much solid is added, the compact mixture disintegrates and therefore cannot be processed further, and in the case of mixtures with semi-active or active solids in particular, crumbling occurs.

The distribution of highly disperse silicic acids as rheology auxiliaries into organic polymers is described in a brochure from Wacker-Chemie GmbH ("HDK Das Verdickungsmittel" [HDK The Thickener], 1985), but the silicic acid content sought here in the finished formulation is relatively low. During preparation of silicone rubber mixtures, to improve the mechanical strength of the crosslinked elastomer, it is necessary to incorporate large amounts of chiefly active solids, such as highly disperse silicic acids, precipitated or naturally occurring chalks or highly disperse metal oxides, such as aluminum oxide or titanium dioxide, into silicone polymers.

It is known to accelerate the incorporation of solids by addition of incorporation auxiliaries. For the preparation of silicone rubber mixtures having a filler content of silicic acid, for example, monomeric or oligomeric silanes which are reactive in respect of the silicic acid surface, such as hexamethyldisilazane or short-chain polydimethylsiloxanes having hydroxyl end groups, are incorporated as auxiliaries; the silicic acid surface is simultaneously hydrophobized, i.e. subjected to a chemical treatment (NOLL "Chemie und Technologie der Silicone" [Chemistry and Technology of the Silicones] 1968, p. 346). The process of in situ hydrophobization of silicic acid with hexamethyldisilazane has been used for a long time for

the preparation of two-component silicone rubbers which crosslink at room temperature and have a filler content of silicic acid (cf., for example, German Patent application DE 24 33 697). In spite of the use of these known auxiliaries and the use of the so-called masterbatch process, the processes known to date impose limits on increasing the silicic acid proportion in the mixture and therefore the intensity of the stress on the goods to be mixed. If other solids are employed, it is possible to employ dispersing auxiliaries. Only relatively small amounts are used here, and these as a rule remain in the product. However, limits are also imposed here on increasing the proportion of solids in the mixture.

DE 25 35 334 describes a process for the homogeneous distribution of highly disperse fillers into polysiloxanes. In this process, the solid is hydrophobized in situ while being mixed into the polymer, and the mixture is then subjected to a mechanical treatment. A silicic acid proportion of about 25% by weight is achieved.

An extrudable material comprising polymer and filler is described in DE 44 42 871. In this case, up to 33 to about 65% by weight of solid is combined with a polymer under conditions of a relatively high shear action, and the components are then mixed, while retaining the high shear forces. In this process, it is already necessary to employ a high shear action during mixing of the filler into the polymer, and therefore an appropriate mixing unit. This can be realized technologically only with difficulty, since high shear forces must already be introduced during the incorporation into the mixture, which is still a relatively thin liquid at this point. A more effective introduction of energy is possible only in mixtures having a higher filler content.

No processes are known which describe the preparation of silicone polymer/solid premixes having a very high filler content in simple mixing units.

SUMMARY OF THE INVENTION

An object of the invention is to increase the proportion of solids in silicone polymer/solid premixes. Using the premixes having a high filler content, it is possible to prepare silicone rubber mixtures which are distinguished by improved flow properties of the non-crosslinked mixtures and very good mechanical properties, such as, a higher extensibility and lower Shore A Hardness of the cured elastomers.

The invention relates to a process for increasing the proportion of solids in silicone polymer/solid premixes and the associated better distribution of the solid in a preferably linear polyorganosiloxane with the aim of improving both the rheological properties of the non-crosslinked mixtures and the elastomer characteristic values.

The proportion of solids is increased according to the invention by a procedure in which a readily volatile siloxane which contains no hydrolyzable groups is first added for incorporation of the solid into a polyorganosiloxane, and is then removed again by means of a vacuum and/or by elevated temperature. As a result, it is possible to increase the proportion of solids in silicone polymer premixes far beyond the maximum proportion in filler/polymer mixtures which can be achieved by processes known to date, without the mixture disintegrating.

These objects are achieved by a process comprising incorporating into a mixture (A) of (a) 50 to 100 parts by weight of at least one polyorganosiloxane containing up to 5 mol % crosslinkable groups, and (b) 5 to 200 parts by weight, preferably 20 to 100 parts by weight, of at least one readily volatile organosilicon compound containing no hydrolyzable groups, 100 to 500 parts by weight, preferably 150 to 300 parts by weight, of a solid (B). The

compound (b) is removed and the mixture having a high filler content is then subjected to mechanical stress.

DETAILED DESCRIPTION OF THE INVENTION

Polyorganosiloxanes (a) which are employed are linear and/or branched polyorganosiloxanes in which up to 5 mol % of the organo radicals are crosslinkable groups and which preferably have more than 50 Si atoms and viscosities of between 0.05 and 100 Pas. Examples of crosslinkable groups are hydroxyl and/or alkoxy groups in condensation-crosslinking systems, dimethylvinylsiloxyl and/or hydrogen end groups and/or hydrogen and/or vinyl side groups in addition-crosslinking systems, or vinyl groups in peroxidically crosslinking systems. Methyl, phenyl and/or trifluoropropyl radicals, *inter alia*, can be bonded as further groups which do not have a crosslinking action. Branched polyorganosiloxanes which are employed are liquid siloxanes which, in addition to di- and monofunctional siloxyl units, also contain tri- and tetrafunctional units. The ratio of tri- and tetrafunctional to monofunctional groups is usually chosen here such that the polymers are liquids at room temperature (25°C). They are used, for example, with the aim of achieving specific flow properties in the elastomers. Mixtures of different polyorganosiloxanes may also be employed. Preferably, the polyorganosiloxanes (a) have a molecular weight of at least 1000.

Readily volatile organosilicon compounds (b) which contain no hydrolyzable groups are those which contain no SiX groupings, in which X is a hydrolyzable radical, for example, hydroxyl, alkoxy, chlorine, oxime, nitrogen or hydrogen radicals. A hexaalkyldisiloxane, further low molecular weight dialkylsiloxanes having up to 5 Si atoms and/or one or more

cyclic dialkyl siloxanes having 3 to 6 silicon atoms are preferably employed. The alkyl groups may contain 1-10 carbon atoms. Advantageously, and for reasons of industrial availability, the compounds employed contain methyl groups as the alkyl groups. The readily volatile organosilicon compound (b) has a low molecular weight, preferably at most 1000. Readily volatile means that at least 99 weight % of the organosilicon compound (b) can be removed from the mixture at a temperature of at most 200°C at a pressure of at least 1 torr in 24 hours or less. No hydrolyzable groups means that less than 1 mol% of the groups bounded to silicon will hydrolyze when the compound is mixed with water at room temperature, in 24 hours.

Solids (B) which can be employed are all the solids known for use in silicone rubber mixtures, in particular active solids, such as highly disperse silicic acids, precipitated or naturally occurring chalks, kieselguhrs or highly disperse metal oxides, such as aluminum oxide or titanium dioxide. Pyrogenic and precipitated silicic acids, for example having a BET surface area of greater than 50 m²/g, are particularly preferred. The surfaces of the solids used may be pretreated.

If highly disperse hydrophilic silicic acid is used, it is advantageous to add, before or during incorporation thereof, 0.1 to 50 parts by weight, preferably 1 to 10 parts by weight, of at least one reactive compound (aa) which is suitable for surface treatment of the silicic acid, the sum of all the parts by weight of mixture (A) being 100. Organosilicon compounds having 1 to 2 Si atoms, for example silazanes, such as hexamethyldisilazane, or trialkylsilanols, for example trimethylsilanol, are preferably used. If silazanes are used, it is customary to work in the presence of water if the moisture adhering to the silicic acid is not sufficient. If silanols are employed, it is favorable to add silazanes or an NH₃/water mixture. Short-chain siloxanes

having up to 10 Si atoms and alkoxy, acetoxy, chlorine and/or hydroxyl groups can furthermore be employed as reactive compounds. Any readily volatile compounds formed are removed from the premix together with compound (b), after the silicic acid has been incorporated.

The incorporation of solids (B) into mixture (A) can be carried out, for example, discontinuously in a twin-shaft kneader, compression mixer, dissolver or in other known mixing units. The process according to the invention may of course also be carried out in continuously operating mixing units, such as a twin-screw extruder. The incorporation may be carried out at room temperature or elevated temperature and under pressures above atmospheric pressure.

After the incorporation, compound (B) and, if appropriate, readily volatile compounds contained in the premix or formed, for example, during the hydrophobization, are removed from the premix, usually by means of a vacuum and/or an increase in temperature.

The resulting mixture having a high filler content is then subjected to mechanical stress by a rotating mixing organ, preferably in the same mixing unit in which the solid has also been mixed in, for example a twin-shaft kneader. The time that the mixture is subjected to stress can last from a few minutes to several hours, and like the intensity of the stress, depends on the desired degree of mixing and the particle size sought for the solid. The increased proportion of solids here allows for considerably more intensive introduction of energy into the premix than is possible with previously known processes. As a result, a considerably better distribution of the solid in the polyorganosiloxane can be achieved.

The mechanical stress can be controlled via the speed of rotation of the mixing organ, the highest possible speed of rotation being sought. As a result of the high proportion of solid

in the premix, however, it is also possible to incorporate the desired kneading and/or shear energy in mixing units with a slowly rotating mixing organ. If a completely filled mixing chamber (free from dead space) is used, it is possible to achieve average particle sizes of silicic acid of less than 100 nm, for example less than 50 nm, as a result of the intensive mechanical stress exerted here on the premix having a high filler content. Mechanical stress under the action of pressure is also possible. The use of a mixing unit which can be heated is also favorable, in order to remove any readily volatile compounds still contained in the mixture from the mixture during or after the mixing operation.

Depending on the later intended use, the premixes having a high filler content obtained by the procedure according to the invention can either be diluted with polyorganosiloxane (a) and/or stored as a silicone rubber premix, or be processed to the ready-cured elastomer, if appropriate after addition of further customary mixture constituents, such as crosslinking agents, plasticizers, catalysts, adhesion promoters, pigments, compounds having a reinforcing action, such as MQ silicone resins, and further active or inactive fillers. To achieve a homogeneous and storage-stable mixture, the dilution is preferably carried out at temperatures above 100°C.

Silicone rubber mixtures which result from the premixes prepared according to the invention have the great advantage over conventional mixtures in that they are exceptionally storage-stable, i.e. retain their viscosity even over several months, and can also be prepared as mixtures having a high filler content and good flow properties. Furthermore, they are of considerably lower viscosity than conventionally prepared mixtures, while retaining good mechanical characteristic values of the elastomers.

The silicone polymer/solid premixes prepared according to the invention can be

employed, for example, in silicone rubber mixtures with good flow properties, as impression and embedding compositions and, if chalk is used, as a solid for the preparation of sealing compositions.

The chemistry of silicones, and the compounds used for their preparation are well known to those of ordinary skill in the art, and are described in "The Encyclopedia of Chemical Technology" Kirk-Othmer, 4th ed., Vol. 22, pages 1-154 (1997, John Wiley & Sons, Inc.), hereby incorporated by reference.

Having generally described this invention, a further understanding can be obtained by reference to certain specific examples which are provided herein for purposes of illustration only and are not intended to be limiting unless otherwise specified.

Embodiment Examples

All viscosity data are based on 25°C.

Example 1

90g of a pyrogenic silicic acid which has not been pretreated and has a surface area of 200 m²/g were incorporated into a mixture of 120 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 5 Pas, 15 g of hexamethyldisiloxane, 24 g of hexamethyldisilazane and 6 g of water in a twin-shaft kneader of 0.5 l capacity. This mixture was kneaded at 130°C for 1 hour. It was then heated up to 160°C in vacuo, the hexamethyldisiloxane and readily volatile compounds formed being removed. The silicone rubber premix having a high filler content thus obtained was kneaded at temperatures between 130 and 160°C for a further hour. It was then rediluted with 120 g of the

polydimethylsiloxane having hydroxyl end groups and 60 g of a polydimethylsiloxane having trimethylsiloxy end groups and a viscosity of 0.1 Pas. The viscosity of the resulting mixture was 34 Pas.

100 g of the mixture prepared was cured at room temperature for 7 days with 5 g of a crosslinking agent liquid which comprised 60 parts by weight of tetraethoxysilane, 35 parts by weight of methyltriethoxysilane and 5 parts by weight of dibutyltin dilaurate. The cured elastomer had a hardness of 31 Shore A and a tear propagation resistance of 25 N/mm.

Example 2

Example 1 was repeated, with the modification that the silicic acid was incorporated into a mixture of 90 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 5 Pas, 45 g of hexamethyldisiloxane, 24 g of hexamethyldisilazane and 6 g of water. The viscosity of the diluted mixture was 27 Pas. The material cured with the crosslinking agent liquid had a hardness of 28 Shore A and a tear propagation resistance of 24 N/mm.

Example 3 (comparative example)

Example 1 was repeated, with the modification that the silicic acid was incorporated into a mixture of 135 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 5 Pas, 24 g of hexamethyldisilazane and 6 g of water. No hexamethyldisiloxane was added. The viscosity of the diluted mixture was 59 Pas. The material cured with the crosslinking agent liquid had a hardness of 29 Shore A and a tear propagation resistance of 25 N/mm.

The comparative example shows that the mixture, which has the same composition as that of Examples 1 and 2, has a considerably higher viscosity which is undesirable for later use, with the same high tear propagation resistance.

Example 4

Example 1 was repeated, with the modification that the silicic acid had a surface area of 300 m²/g and was incorporated into a mixture of 65 g of a polydimethylsiloxane having dimethylvinylsiloxo end groups and a viscosity of 6 Pas, 45 g of hexamethyldisiloxane, 28.5 g of hexamethyldisilazane, 1.5 g of divinyltetramethyldisilazane and 7.5 g of water, and the mixture was kneaded at 130°C for 1 hour. The resulting mixture was diluted with 145 g of polydimethylsiloxane having dimethylvinylsiloxo end groups and a viscosity of 6 Pas. The viscosity of the resulting mixture was 172 Pas.

90 g of the mixture were cured at 150°C for 30 minutes with a crosslinking agent component comprising 6.9 g of polydimethylsiloxane having dimethylvinylsiloxo end groups and a viscosity of 6 Pas, 0.3 g of a 1% strength Pt catalyst and 2.8 g of polydimethylsiloxane having dimethylhydrosiloxo end groups and an H-Si content of 4.0 mmol/g. The cured material had a hardness of 28 Shore A and a tear propagation resistance of 30 N/mm.

Example 5 (comparative example)

Example 4 was repeated, with the modification that no hexamethyldisiloxane was incorporated. The viscosity of the diluted mixture was 272 Pas. The material cured with the crosslinking agent component according to Example 4 had a hardness of 29 Shore A and a tear propagation resistance of 30 N/mm.

The comparative example without addition of hexamethyldisilazane shows that the finished mixture which has the same composition as that from Example 4 has a considerably higher viscosity which is undesirable for later use, with the same high hardness and tear propagation resistance.

Example 6

Example 4 was repeated, with the modification that the silicic acid was incorporated into a mixture of 65 g of a polydimethylsiloxane having dimethylvinylsiloxo end groups and a viscosity of 60 Pas, 45 g of octamethylcyclotetrasiloxane, 28.5 g of hexamethyldisilazane, 1.5 g of divinyltetramethyldisilazane and 7.5 g of water. Redilution was carried out with 295 g of the polydimethylsiloxane having dimethylvinylsiloxo end groups and a viscosity of 60 Pas. The viscosity of the resulting mixture was 239 Pas.

90 g of this mixture was cured at 150°C for 30 minutes with a crosslinking agent component comprising 8.3 g of polydimethylsiloxane having dimethylvinylsiloxo end groups and a viscosity of 6 Pas, 0.3 g of a 1% strength Pt catalyst and 1 g of polydimethylsiloxane having dimethylhydrogensiloxo end groups and an H-Si content of 7.0 mmol/g. The cured material had a hardness of 30 Shore A and a tear propagation resistance of 20 N/mm.

Example 7

Example 1 was repeated, with the modification that the resulting silicone rubber premix having a high filler content was subjected to intensive mechanical stress in a laboratory compression mixer with no dead space at a speed of rotation of the mixing organ of 10 ms⁻¹ under a pressure of 9 bar and at a temperature of 120°C for 120 minutes. Before

further dilution, an average particle size of the filler of 40 nm was determined by means of transmission electron microscopy. The viscosity of the diluted mixture was 28 Pas.

Example 8

Example 1 was repeated, with the modification that after the exposure to mechanical stress, the mixture having a high filler content was diluted at a temperature of 120°C. The viscosity of the resulting mixture was 32 Pas.

Example 9

270 g of a precipitated chalk treated with stearic acid and having a surface area of 4 m²/g were incorporated into a mixture of 90 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 30 Pas, 6 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 0.05 Pas, 30 g of a polydimethylsiloxane having trimethylsiloxy end groups and a viscosity of 0.1 Pas and 60 g of hexamethyldisiloxane in a twinshaft kneader of 0.5 l capacity. This mixture was heated up to 110°C in vacuo, with constant kneading, the non-reactive hexamethyldisiloxane being removed from the mixture. The silicone rubber premix having a high filler content thus obtained was then kneaded at 80°C for one hour. After the mechanical stress, the mixture was rediluted with a further 180 g of the polydimethylsiloxane having hydroxyl end groups and a viscosity of 30 Pas. The viscosity of the diluted mixture was 129 Pas.

100 g of the resulting mixture were cured at room temperature for 7 days with 10 g of a crosslinking agent liquid comprising 20 parts by weight of a partial hydrolyzate of methyltrimethoxysilane, 10 parts by weight of propyltriethoxysilane, 55 parts by weight of an

adhesion promoter combination of aminopropyltriethoxysilane and glycidoxytrimethoxysilane, 10 parts by weight of carbon black and 5 parts by weight of dibutyltin dilaurate. The cured material had a hardness of 52 Shore A. Glass test specimens produced from the material had an elongation at break of 120% and a tensile strength of 1.0 MPa.

Example 10

Example 9 was repeated, with the modification that 270 g of a precipitated chalk treated with stearic acid and having a surface area of 4 m²/g were incorporated into a mixture of 60 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 30 Pas, 6 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 0.05 Pas, 30 g of a polydimethylsiloxane having trimethylsiloxy end groups and a viscosity of 0.1 Pas and 90 g of hexamethyldisiloxane. After the mechanical stress, the mixture was rediluted with a further 210 g of the polydimethylsiloxane having hydroxyl end groups and a viscosity of 30 Pas. The viscosity of the resulting mixture was 124 Pas.

100 g of the finished mixture were cured at room temperature for 7 days with 10 g of the crosslinking agent liquid. The cured material had a hardness of 50 Shore A. An elongation at break of 150% and a tensile strength of 1.1 MPa were measured on glass test specimens.

Example 11 (comparative example)

Example 9 was repeated, with the modification that 270 g of a precipitated chalk treated with stearic acid and having a surface area of 4 m²/g were incorporated into a mixture

of 150 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 30 Pas, 6 g of a polydimethylsiloxane having hydroxyl end groups and a viscosity of 0.05 Pas and 30 g of a polydimethylsiloxane having trimethylsiloxy end groups and a viscosity of 0.1 Pas. After kneading at 80°C for 1 hour, the resulting mixture was rediluted with a further 210 g of the polydimethylsiloxane having hydroxyl end groups and a viscosity of 30 Pas. The viscosity of the resulting mixture was 182 Pas.

100 g of the finished mixture were cured at room temperature for 7 days with 10 g of the crosslinking agent liquid. The cured material had a hardness of 56 Shore A. An elongation at break of 110% and a tensile strength of 0.9 MPa were measured on glass test specimens.

Comparative example 11 shows that the mixture, which has the same composition as that of Examples 4 and 5, has a high viscosity which is undesirable for later use. The glass test specimens produced with this mixture do not achieve sufficiently high tensile strengths and elongations at break.

Obviously, additional modifications and variations of the present invention are possible in light of the above teachings. It is therefore to be understood that within the scope of the appended claims, the invention may be practiced otherwise than as specifically described herein.

The priority document of the present application, German Patent Application No. DE 196 53 993, filed December 21, 1996, is hereby incorporated by reference.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:-

1. A process for the preparation of a silicone polymer/solid premix, comprising:
incorporating into (A) a mixture comprising
 - (a) 50 to 100 parts by weight of at least one polyorganosiloxane containing up to 5 mol % of crosslinkable groups, and
 - (b) 5 to 200 parts by weight of at least one readily volatile organosilicon compound containing no hydrolyzable groups,100 to 500 parts by weight of (B) a solid, thereby forming a composition;
removing (b) said organosilicon compound from said composition; and then
subjecting said composition to mechanical stress.
2. The process of Claim 1, wherein (a) said polyorganosiloxane has a molecular weight of at least 1000.
3. The process of Claim 1, wherein (a) said polyorganosiloxane contains at least one member selected from the group consisting of dimethylhydroxysiloxy, trimethylsiloxy and dimethylvinylsiloxy end groups.
4. The process of Claim 1, wherein (b) said readily volatile organosilicon compound is at least one member selected from the group consisting of hexaalkyldisiloxanes, octaalkyltrisiloxanes, and cyclic dialkylsiloxanes containing 3 to 6 silicon atoms.
5. The process of Claim 1, wherein (B) said solid is chalk.
6. The process of Claim 1, wherein (B) said solid is highly disperse silicic acid.

7. The process of Claim 1, wherein (A) said mixture further comprises (c) 0.1 to 50 parts by weight of at least one compound suitable for surface treatment of highly disperse silicic acid, and

(B) said solid is silicic acid.

8. The process of Claim 7, wherein (c) said compound is selected from the group consisting of disilazanes and trialkylsilanols.

9. The process of Claim 3, wherein (b) said readily volatile organosilicon compound is at least one member selected from the group consisting of hexaalkyldisiloxanes, octaalkyltrisiloxanes, and cyclic dialkylsiloxanes containing 3 to 6 silicon atoms.

10. The process of Claim 9, wherein (c) said compound is selected from the group consisting of disilazanes and trialkylsilanols.

11. The product produced by the process of Claim 1.

12. The product produced by the process of Claim 4.

13. The product produced by the process of Claim 10.

14. A process for the preparation of an intermediate composition, comprising:
incorporating into (A) a mixture comprising

(a) 50 to 100 parts by weight of at least one polyorganosiloxane containing up to 5 mol % of crosslinkable groups, and

(b) 5 to 200 parts by weight of at least one readily volatile organosilicon compound containing no hydrolyzable groups,

100 to 500 parts by weight of (B) a solid, thereby forming said composition.

15. The process of Claim 14, wherein (a) said polyorganosiloxane contains at least one member selected from the group consisting of dimethylhydroxysiloxy, trimethylsiloxy and dimethylvinylsiloxy end groups.

16. The process of Claim 14, wherein (b) said readily volatile organosilicon compound is at least one member selected from the group consisting of hexaalkyldisiloxanes, octaalkyltrisiloxanes, and cyclic dialkylsiloxanes containing 3 to 6 silicon atoms.

17. The product produced by the process of Claim 14.

18. The process of Claim 1, further comprising, after said subjecting, curing said composition.

19. The process of Claim 10, further comprising, after said subjecting, curing said composition.

20. The product produced by the process of Claim 18.

Process for the production of highly filled silicone polymer masterbatches

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Abstract

Production of highly filled silicone polymer-solid pre-mix comprises (a) working 100-500 parts weight (pts. wt.) solid (I) into a mixture of 100 pts. wt. linear and/or branched polyorganosiloxane(s) (II), in which ≤ 5 mole-% of the organo groups can be crosslinked, and 5-200 pts. wt. readily volatile, low-molecular organosilicon compound (III) containing no hydrolysable groups; (b) removing (III); and (c) subjecting the (II)-(I) mixture to mechanical stress.

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